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FINAL REPORT ON THE EVALUATION OF CHEMICAL MILLING AS A POSSIBLE PROCESS TECHNIQUE IN THE PRODUCTION OF ½-HARD AND 3/4-HARD 301 STAINLESS STEEL BULKHEADS

MRG-160

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SUBJECT: Final Report on the Evaluation of Chemical Milling as a Possible Process Technique in the Production of ½- Hard and 3/4- Hard 301 Stainless Steel Bulkheads.

ABSTRACT

The effect of the chemical milling process on the tensile and fatigue properties, corrosion resistance, thickness tolerance, etc., of base metal and weld joints of stretch-formed Type 301 cold worked stainless steel sheet has been evaluated. Three major deterrents to the use of chemical milling as a fabrication technique were established by this work. First, the fatigue properties of the base metal and the heliarc weld joints appeared to be considerably reduced. Second, hydrogen embrittlement was found to exist in the metal after the chem-milling process and this could not be completely relieved without resorting to a vacuum degassing treatment. Third, corrosion resistance was decreased markedly. Thickness tolerances were kept within 0.001 inch with a surface finish between 60 and 120 rms. A preferential acid attack resulted in undercutting of the weld area. However, this could be overcome by appropriate masking techniques.

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SUBJECT: Final Report on the Evaluation of Chemical Milling as a Possible Process Technique in the Production of ½- Hard and 3/4-Hard 301 Stainless Steel Bulkheads.

INTRODUCTION

At the request of the Structural Design Group, a study was initiated to evaluate the effects of chemical milling on the mechanical properties, corrosion resistance, surface finish, thickness tolerance, etc., of ½-H and 3/4-H 301 stainless steel bulkhead sections.

In order to evaluate this processing technique correctly, it was decided to have the chemical milling performed by a reputable vendor rather than attempt a laboratory simulation of the process. Although this has led to considerable delay, it is by far the most satisfactory approach.

A progress report on this evaluation program was issued on 29 March 1960 (Report No. MRG-144). The results of thickness tolerance and surface finish measurements visual, x-ray and metallographic examinations as well as hydrogen embrittlement studies were presented.

It is the purpose of this report to present the results of the remainder of the program. These are concerned with mechanical properties such as tensile and fatigue, corrosion properties and means of relieving hydrogen embrittlement.

However, before proceeding to the results and discussion of the current phases of the program it would be well to review the major conclusions presented in the first progress report. These are as follows:

1. In general, thickness variations are increased by chemical milling but it appears that these variations can be kept on within 0.001 inch in the present case.

2. The surface finish was decreased from 17 rms as stretched-formed to between 60 and 120 rms after chemical milling.

3. Careful examination indicated that no unusual chemical attack occurred in the areas of high Lüeder line areas.

4. X-ray examination showed that the weld areas were sound before and after chemical milling.

5. After a 50-percent reduction in base metal thickness by chemical milling, the thickness variation in the nugget was increased. Furthermore, a selective attack was also noted in the heat-affected zones. After chemical milling, the heat-affected zones were thinner than the base metal by about 9 percent. The minimum thickness in the nugget area was as much as 27 percent thinner than the base metal.

6. It was found that this material was hydrogen embrittled by the chemical milling process, but the severity of this embrittlement could not be determined.

Obviously, the two major effects of chemical milling uncovered during the initial phase of this study are associated with the selective attack of the weld nugget and heat-affected zones and the introduction of hydrogen embrittlement. It was suggested that it is possible to overcome the thinning problem (selective attack of the heat-affected zone and weld nugget) by removing the bulkhead sections from the chemical milling solution when a few thousandths of metal remain to be removed. The weld area could then be masked and the remaining metal removed from the bulkheads.

Techniques for decreasing the severity of hydrogen embrittlement were established during this phase of the study and will be reported on in the sections that follow.

RESULTS AND DISCUSSION

Partial bulkhead sections made from stretch-formed ½- Hard and 3/4- Hard 301 stainless steel were chemically milled from 0.020 (or 0.025) inch thickness to 0.010 inch by the United States Chemical Milling Corporation. Standard tensile coupons were machined from both bulkhead sections. Both base metal and weld coupons were obtained. Samples of the chemically milled bulkheads 3" x 3" were removed for salt spray corrosion tests.

TENSILE TESTS

Tensile tests were performed at room temperatures. Tests were run on samples machined from the weld areas such that the heliarc butt weld was in the center of the reduced section and was perpendicular to the length of the tensile coupon. Samples for base metal property evaluation were also obtained from the bulkhead sections away from the welded area but in the same direction as the weld test coupon. Triplicate samples were prepared and tested.

As shown in Table 1, the tensile properties were essentially unaffected by the chemical milling process. The small variations that are observed can be attributed to several factors. The unchem-milled samples were 0.020 inch thick while the chemical milled test coupons were only 0.010 inch. Over and above the thickness effects that may be introduced, normal scatter and test accuracy would account for most if not all of the differences observed, especially on samples taken from a stretch-formed bulkhead section.

FATIGUE TESTS

Extra samples had been machined for the tensile test phase of this study. Because they were not needed for tensile testing it was decided to use them for a preliminary evaluation of fatigue properties. Since these coupons were not the best specimen configuration for a fatigue test, it was anticipated that some trouble might be experienced in the testing phase. This proved to be true since some of the coupons failed at one end of the reduced section or the other instead of in the central portion for which accurate dimensions were known. In spite of this problem the data of Table 2 appear to show some significant trends.

The data of Table 2 were obtained from tension-tension tests on a Baldwin-Tate-Emery (SF-1-U) fatigue tester. In general the data presented are the average of duplicate tests, but in some cases triplicate tests were obtained. As in the case of tensile testing, the properties of the chemically milled bulkheads were developed on samples 0.010 inch thick whereas the original metal properties were obtained on metal of .020(or .025) inch thickness. Alignment and dimensional measurements of the thin samples were very difficult to attain accurately, especially in the weld areas. Despite the problems of alignment, dimensional accuracy, and small sample size, the large decrease in the number of cycles to failure after chemical milling strongly suggests that fatigue properties are indeed effected adversely by the chem-mill process.

CORROSION RESISTANCE

The corrosion resistance of the chemical milled surface was not known and it appeared that a preliminary investigation of this problem should be undertaken. Test panels as shown below were subjected to salt spray tests for 1000 hours duration:

1. Control - surface as-stretch-formed

2. Chemically-milled

3. Chemically-milled plus passivated in HNO_3 solution

As expected, the control panel survived this test readily with only a few easily removed surface stains being observed. The chemically milled surfaces showed a more general staining but this too was easily removed.

However, two or three isolated points which were severely attacked were noted. In these areas, corrosion resulted in complete penetration of the 0.010 sheet and a small pin hole was formed. This pin hole corrosion occurred on the passivated samples as well as the unpassivated sample.

HYDROGEN EMBRITTLEMENT

Because of the thinness of the bulkhead sections hydrogen embrittlement tests could not be performed on this material. However, samples of 0.060 full hard 301 stainless steel were submitted to the vendor along with the bulkhead sections. The vendor chem-milled these samples to 0.040 and returned them for test. As mentioned in the first report, these samples were embrittled by the chem-mill process.

Hydrogen embrittlement of heat-treated low alloy steels can be relieved by a low temperature baking treatment and consequently, processes which introduce embrittlement in these steels can be used if the materials are baked after the process has been employed.

Attempts were made to eliminate the hydrogen embrittlement of 301 stainless steel introduced by the chem-mill process by various baking treatments. It proved impossible to completely eliminate the hydrogen embrittlement even at temperatures up to 500°F. It was felt that higher baking temperatures might affect the base metal properties. Therefore, vacuum degassing treatments were investigated as a possible means of eliminating the hydrogen. It was found that vacuum degassing at room temperature with a relatively poor vacuum (30-50 microns) completely eliminated the hydrogen embrittlement introduced from the chemical milling process.

Unfortunately, it is not possible to specify at this time what degree of hydrogen embrittlement can be tolerated in the 301 stainless steel without leading to catastrophic failure. Vacuum degassing of 10' diameter bulkheads is not a desirable or even very feasible production process. An 8 hour bake at 400°F could be carried out but some degree of hydrogen embrittlement will remain.

CONCLUSIONS

Based on a limited amount of data, the following conclusions can be drawn regarding the effect of chemical milling on the static, fatigue, and corrosion properties of ½ and 3/4 hard 301 stainless steel bulkhead material.

1. Tensile properties (F_{tu}, F_{ty}, E, e) remain essentially unaffected.

2. Fatigue properties (cycles to failure) appear to be reduced considerably.

3. General resistance to corrosion is reduced slightly but the

resistance to localized attack such as pin-hole corrosion is markedly reduced.

4. Hydrogen embrittlement, introduced during the milling process can be reduced considerably by a baking treatment of 8 hours at 400°F or completely eliminated by vacuum degassing at room temperatures.

RECOMMENDATIONS

The results of this investigation show that chemical milling affects the properties of 301 bulkhead material in several respects. Before using materials processed in this manner a complete evaluation of these effects are necessary, since design allowables may be changed considerably, especially fatigue allowables.

Because of the undue amount of testing necessary to establish new design allowables on 301 stainless processed by the chem-milling technique, it is recommended that the process be used only when it is not possible to obtain the final product by more conventional means.

If it is necessary to use the chem-mill process, further investigation of fatigue properties, corrosion resistance and hydrogen embrittlement is strongly recommended. However, the deleterious effects of this process upon the fatigue and corrosion resistance of cold worked and stretch-formed Type 301 stainless steel sheet would indicate that other methods of effecting weight reductions should be more thoroughly investigated before chem-milling is selected as the production process.

Table 1

TENSILE DATA AT ROOM TEMPERATURE FOR 301 STAINLESS STEEL BULKHEADS

BEFORE AND AFTER CHEMICAL MILLING

Material	F_{tu}	F_{ty}	e	E
Prior to Chem-Mill	psi	psi	percent	psi
½ Hard Base Metal	187,000	126,000	20.7	25.9×10^6
½ Hard Weld Metal	127,000	-		
3/4 Hard Base Metal	204,000	170,000	5.0	26.5×10^6
3/4 Hard Weld Metal	138,000	-		
After Chem-Mill				
½ Hard Base Metal	189,000	158,000	14.7	24.4×10^6
½ Hard Weld Metal	138,000	-		
3/4 Hard Base Metal	201,000	169,000	6.5	24.6×10^6
3/4 Hard Weld Metal	135,000	-		

Table 2

FATIGUE DATA AT ROOM TEMPERATURE FOR 301 STAINLESS STEEL BULKHEAD SECTIONS BEFORE AND AFTER CHEMICAL MILLING

MATERIAL	STRESS LEVEL TENSION-TENSION FATIGUE	CYCLES TO FAILURE
Prior to Chem-Mill		
½ Hard - Base	120,000 psi max.-10,000 psi min.	160,000
½ Hard - Weld	80,000 psi max.-10,000 psi min.	201,000
3/4 Hard - Base	130,000 psi max.-10,000 psi min.	86,000
3/4 Hard - Weld	80,000 psi max.-10,000 psi min.	148,000
After Chem-Mill		
½ Hard - Base	120,000 psi max.-10,000 psi min.	80,000
½ Hard - Weld	80,000 psi max.-10,000 psi min.	16,000
3/4 Hard - Base	130,000 psi max.-10,000 psi min.	30,000
3/4 Hard - Weld	80,000 psi max.-10,000 psi min.	36,000

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